Syllabus MSNE 437+537 revised 10Jan2020 PJL

CRYSTALLOGRAPHY AND DIFFRACTION LAB - Selected lab experiments in materials science. This course is designed to complement the lectures in MSNE/PHYS 435/535, which must be taken concurrently or previously. The lab experiments concern the structure and constitution of materials as revealed by x-ray scattering, including both diffraction and refraction. **This lab is required for the BS MSNE degree** but optional for other degrees. Undergrads planning to stay at Rice for grad school should probably take 537.

Instructor: Dr. Peter Loos **office**: MEB 215 ploos@rice.edu **Office hours:** after class and by appointment.

Teaching Assistants: **X-ray Lab Manager:** Dr. Jianhua Li jianhua.li@rice.edu

Location: Experimental work is done in Butcher 160. Some sample preparation may occur in Ryon B08.

Schedule: TBD, perhaps section meetings Tuesday 2:30 & 4:00 and/or Wednesday 11:00 & 1:00.

Text: Cullity, ***Elements of X-ray Diffraction***, either 3rd or 2nd edition but **not** the 1956 1st edition. Other reading will also be assigned.

Grading: Based on attendance, participation, safety, neatness, ability to operate the equipment, and accuracy of results turned in. Students whose efforts during the semester are unsatisfactory will have the opportunity to take a final exam for extra credit.

437 students will complete at least seven of the lab exercises. **537 students** will complete at least eight.

Absence Policy: Students are expected to attend every class. Contact Instructor and/or TA as soon as it becomes apparent that an absence cannot be avoided. Any absence from the hands-on data collection must be made up.

Cell Phones: These can be used for photographing samples and equipment but otherwise will be put away and out of sight.

Safety: There will be a reading assignment and quiz about x-ray safety. The quiz can be taken as many times as needed to get all the answers correct.

Areas of Instruction: Selected lab exercises from the list below will be accompanied by reading in the text and in other references. These exercises may be selected or changed based on interests expressed by the students and on availability of the equipment.

#1 X-ray safety
#2 Instrument 2θ Alignment & Calibration, detector optimization using NIST SRM 640D silicon.
#3 Film thickness, density & roughness by x-ray reflectometry/reflectivity (XRR) which is neither crystallography nor diffraction.
#4 Emission spectrum of the Cu x-ray tube, x-ray absorption, filtering the spectrum.
#5 Identification of unknown powder samples via search/match of the ICDD Powder Diffraction File (PDF) using Jade or PDXL.
#6 Determination of volume fractions of multiple phases in a mixture using reference intensity ratios.
#7 Crystallite size and microstrain determined from XRD peak broadening using Scherrer's formula and a Williamson-Hall plot
#8 Precise lattice parameter measurement and composition of solid solutions using Vegard's Law and a spreadsheet.
#9 Identifying unknown samples and indexing their diffractometer patterns using Jade or PDXL and PDF.
#10 Measurement of the fractions which are crystalline and amorphous in PTFE polymer
#11 Examination & identification of nanomaterials used as corrosion inhibitors in a hydrocarbon-based lubricant
#12 Preferred orientation (texture) in crystalline material using pole figures
#13 Phase changes in perovskite as a function of temperature (using a new hot stage arriving spring 2020)

Instrumentation: Dmax West is used in experiments #2 & 5-11 but SmartLab is used in experiments #3, 4 & 12. Experiments #5, 6, and 8-11 can also be run on Dmax East or SmartLab if desired or if necessary. These Rigaku instruments are all located in Butcher 160. For more info about them see https://sea.rice.edu/instruments/x-ray-facility. Single crystal XRD instruments available to students include a Rigaku SCXmini small molecule system in Butcher 370 and a Rigaku macro-molecule system in Keck 328.

Lab Results: Students work together in small groups when operating the equipment and collecting & analyzing data with the MDI, Rigaku, and ICDD proprietary softwares; however, students must independently use their own spreadsheets to analyze other experimental data. Results are turned in per the Rice Honor Code and due two weeks after data are collected. Points may be deducted for late results.

Computation: We will use MDI Jade and/or Rigaku PDXL as well as the ICDD Powder Diffraction File (PDF) database. Multiple copies of these are running on Windows machines in Butcher 160. We will also use Rigaku GlobalFit or LBL Bilayer Reflectivity software for reflectivity analysis. Globalfit is installed on two computers in Butcher 160. Spreadsheet software will also be used extensively. Rigaku 3D Explore software is used to produce pole figures. Rigaku data in their proprietary binary RAW format can be reformatted into standard spreadsheet formats using PowDLL or Jade.

Goals for this course: To address ABET Student Outcomes and Program Criteria, students will have an ability to:

1. identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics
6. develop and conduct appropriate experimentation, analyze & interpret data, and use engineering judgment to draw conclusions.

and the course will prepare graduates to:

- apply advanced science, … computational techniques and engineering principles to materials systems …
- utilize experimental, computational and statistical methods to solve materials problems …

More specifically, students will be able to:

(a) determine crystal structure of most metals and ceramics experimentally.
(b) use XRD data to determine composition of binary solid solutions and size of crystallites in a crystalline solid.
(c) use reflectometry to characterize thickness, density, and roughness of thin films.
(d) operate the Rigaku Dmax machines unsupervised.
(e) use many of the features in Jade and/or PDXL software.

NOTICE: Any student with a disability requiring accommodations in this class is encouraged to contact Instructors after class or during office hours. Additionally, students should contact the Disabled Student Services in Allen Center.